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EDGEWOOD ARSENAL TECHNICAL REPORT

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DEVELOPMENT OF ENCAPSULATED WHITE PHOSPHORUS

by

W. Paul Henderson

Manufacturing Technology Directorate

September 1975

U.S. ARMY ARMAMENT COMMAND

DEPARTMENT OF THE ARMY
Headquarters, Edgewood Arsenal
Aberdeen Proving Ground, Maryland 21010

EDGEWOOD ARSENAL

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PREFACE

The work described in this report was conducted as part of a joint program with Picatinny Arsenal, Dover, New Jersey, by direction of COL Irving G. Mollen, Director of Research, Development and Engineering, United States Army Munitions Command, Dover, New Jersey 07801. The work was started on 1 December 1966 and completed on 24 January 1969. The experimental data are contained in the files of Process Technology Branch, Chemical and Plants Division, Manufacturing Technology Directorate.

The use of trade names in this report does not constitute an official endorsement or approval of the use of such commercial hardware or software. This report may not be cited for purposes of advertisement.

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The author wishes to acknowledge the conception of the WP/epoxy system by Mr. George T. Woodward, and the technical assistance rendered by Lieutenant James Mackwell, Mr. Clifford Townsley, Mr. Thomas Lancaster, and Mr. Peter Mirabella.

SUMMARY

White phosphorus (WP) has been incorporated in a variety of munitions for its ability to produce a highly visible and dense smoke for screening and target marking. One serious problem has been encountered when munitions are stored and fired at temperatures above the melting point of WP: the movement of liquid WP within the rounds creates ballistic instability in flight. This study was conducted to develop an optimum WP/polymer system which will improve the ballistic stability of WP rounds at temperatures above 115°F while maintaining the marking and screening properties of the standard WP munition.

CONTENTS

DEVELOPMENT OF ENCAPSULATED WHITE PHOSPHORUS

I. INTRODUCTION.

A. Purpose.

1. To develop a white phosphorus/polymer which will remain solid over the accepted military operating temperatures, thus avoiding the liquid phase and the attendant ballistic stability problems of white phosphorus (WP) munitions at temperatures above the 115°F melting point.

2. To design, fabricate, and operate a prototype pilot facility to process and fill artillery rounds up to eight inches.

B. Background.

The use of white phosphorus has long been recognized for its ability to produce a highly visible and dense smoke for screening and target marking. One serious problem has often been encountered when the munitions, expecially artillery shells, are stored and then fired at temperatures above the melting point of WP. The movement of liquid WP within the shells has created serious ballistic instability in flight. This has plagued developers of WP munitions for years; many approaches have been tried to obtain munitions that are ballistically stable above 115°F. Attempts to overcome this instability have included insertion of fins, baffles, aluminum sponge, small pieces of tubing, rubberized WP, plasticized WP, etc. Generally, there was very little improvement on rounds fired at temperatures above 115°F when using the above techniques.

A letter was received by the Commander, Edgewood Arsenal, MD, from Headquarters, United States Army Munitions Command, Dover, NJ, requesting that Edgewood Arsenal and Picatinny Arsenal prepare a joint program to rapidly exploit the potentials of using WP/epoxy mixture in smoke ammunition.

This report describes the following efforts:

1. Development of a WP/epoxy system.

2. Design, fabrication and operation of a WP/epoxy pilot, batch-type processing and filling system.

3. Investigation of various epoxy-hardener systems.

4. Static and dynamic testing program.

5. Design and fabrication of a prototype, continuous processing and filling system.

II. EXPERIMENTATION AND TEST RESULTS.

A. Development and Testing of a WP/Epoxy Formulation.

The first WP/epoxy mixture formulated was prepared by using a commercially available epoxy-hardener system known as "Fix and Patch." Six 40mm XM574 rounds – each with a 3/8-in filling hole – were filled with a 70% WP and 30% epoxy mixture, using the above epoxy system.

The method of loading and formulation was as follows:

1. WP granules were made by melting solid 2- to 3-pound blocks of material with hot water in a 55-gallon drum. An agitator was used to mix the molten WP with a high shear rate. At this time ice water was

added as agitation continued and granules were formed. A rough sieve analysis was made and the granules used went through 14 mesh and were collected on 30 mesh.

2. The desired quantity of WP was weighed out under water. The epoxy resin was added and mixed by hand with the WP and water. Next, the excess water was poured off and the epoxy hardener was mixed in by hand. Any additional excess water was poured off.

3. The WP/epoxy material was then loaded by hand with a spatula through a 3/8-in diameter filling hole. The munition was then capped with a plastic plug and left to cure overnight. The day after filling, the munition was welded closed. The average weight of the munition fill was 43 grams.

These six rounds were test fired at Aberdeen Proving Ground (APG). All rounds were fired at elevated temperature (130°F) and functioned on target. With the results of this initial firing, the manufacturer of the epoxy (Specialty Plastics, Baltimore) was contacted. The manufacturer is a formulator and uses a base resin and hardener purchased from CIBA. He formulates "Fix and Patch" by adding a filler (which is 68% by weight of the resin and hardener) and a thixotropic agent which he would not reveal. He referred to the resin and hardener as the C-1 formulation.

Guided by the results of the firing tests, the investigators filled further rounds with the C-1 epoxy system and various other resin-hardener systems furnished by Specialty Plastics. Rounds filled were as follows:

1. C-1 Formulation (77 each, 43 grams net weight)

   50 each for test firing at APG
   25 each for transportation-vibration tests at Edgewood Arsenal
   1 each control
   1 each sectioned for inspection

2. WE-WE Formulation (15 each, 43 grams net weight)

   14 each for test firing at APG
   1 each control

3. WE-H-25 Formulation (10 each, 43 grams net weight)

   9 each for test firing at APG
   1 each control

4. R-100-H, 25 Formulation (1:1 ratio, 5 each, 40 grams net weight)

   4 each for testing firing at APG
   1 each control

5. R-100-H, 25 + 10% Excess Formulation (5 each, 40 grams net weight)

   4 each for test firing at APG
   1 each control

6. C-1, L Formulation (5 each, 43 grams net weight)

   4 each for test firing at APG
   1 each control

7. WE, H-25 + Filler Formulation (5 each, 42 grams net weight)

   4 each for test firing at APG
   1 each control

8. R-100, H-25, -10% Formulation (5 each, 40 grams net weight)

   4 each for test firing at APG
   1 each control

In order to make this quantity of WP/epoxy, granulation had to be done on a larger scale. One hundred pounds of solid WP in 2- to 3-pound blocks was melted in a 55-gallon stainless steel drum and brought up to temperature by means of a steam jacket to approximately 155°F. A one-third-horsepower, variable-speed, explosion-proof motor with an 8-in turbine blade was used to agitate the WP-water mixture. While the mixture of molten WP and water was being agitated at approximately 15,000 to 17,000 RPM, ice water was added rapidly until the temperature of the mix reached 86°F at which time the WP had solidified into small particles. A random sampling was made with the following results:

100% passed through 4 mesh
97% passed through 14 mesh
64% passed through 16 mesh
25% passed through 30 mesh

The particles used for mixing the epoxy all went through 14 mesh and were collected on 30 mesh.

The formulation procedure for all the various epoxy systems tried was the same as that previously described, with the following exception: the shells were loaded by using a caulking gun in all cases except with the C-1 formulation which was loaded by hand due to its high viscosity.

The filled rounds were fired at Aberdeen Proving Ground. The results of the firings:

Formulation	Firing Temperature	Number of rounds on target/fired
C-1	Ambient	18/19
C-1	-25°F	10/10
C-1	130°F	10/20
C-1L	130°F	3/4
WE-WE	-25°F	4/4
WE-WE	130°F	0/10
WE-H-25 + Filler	130°F	2/4
WE-H-25	130°F	9/9
R-H-25	130°F	1/4
R-H-25 -10%	130°F	0/4
R-H-25 + 10%	130°F	1/4

With the results of this test and previously obtained data, it was decided to fill 50 rounds each of C-1 formulation and WE-H-25 formulation. Ten additional rounds were filled and closed immediately with no cure time – five rounds with each type of resin. The results of this firing were as follows:

Formulation	Firing Temperature	Number of rounds on target/fired
WE-H-25	130°F	14/47
C-1	130°F	48/48
C-1	130°F (immediate closure)	0/5
WE-H-25	130°F (immediate closure)	0/5

Work on the processing of the WP/epoxy system continued. Initially, the C-1 resin and WP granules were mixed by hand. However, larger batches were required, as was the need for uniform mixing. The needs were met by using a Blakesley (Planetary) Mixer. The next variable in the system which was stabilized was the water content in the WP/epoxy system. Hand-pressing on a 30 mesh screen had been used, leading to a large variation in water content in the final mixture. But with the aid of an air-cylinder, piston arrangement on a 30 mesh screen, it was possible to decant water to a uniform extent in every batch.

With these innovations, the WP/epoxy system formulation was changed as follows:

1. Granulation remained the same as previously mentioned.

2. Epoxy resin and WP granules with water were mixed in the Blakesley mixer.

3. After the mixing operation was completed, the resin-WP mix was placed in the press and water was pressed out.

4. The dried resin-WP was returned to the mixer.

5. The epoxy hardener (or co-polymer) was added and the system was again mixed, using the Blakesley mixer.

At this point a further loading development was made. A loading gun, similar in design to an automotive grease gun, was made of aluminum, with an aluminum piston. The piston was driven by a hydraulic press with a 7-1/2-in diameter ram. A load of 100 psi was required on the 7-1/2-in ram to extrude the C-1, WP system through a 1/4-in diameter hole in a 3/8-in nozzle. The filling hole in the 40mm XM574 round was increased from 1/4-in to 3/8-in diameter.

Using the remaining hardware, 150 rounds, the following tests were run:

System	Test	Number of rounds
C-1	Temperature cycling, vibration test	25
WE, H-25	Temperature cycling, vibration test	25
C-1	Immediate closure	25
WE, H-25	Immediate closure	25
C-1	Dry WP	25
WE, H-25	Dry WP	25

Test Term Definitions:

Temperature cycling. Filled munitions cycled from -65°F to +165°F at regular intervals as prescribed by MIL-STD transportation-vibration test.

Vibration Test. Filled munitions vibrated in various positions as prescribed by MIL-STD transportation-vibration test.

Immediate closure. The closure completed as soon as possible after filling. On this case, this time period ranged from 90 minutes for the first shell filled to 30 minutes for the last shell filled.

Dry WP. The use of WP without water before being mixed with the epoxy resin. Mechanical and chemical methods were investigated.

Several methods of drying WP granules were attempted for the filling of WP/epoxy rounds in a water-free system. A tumble dryer was constructed into which water-wet WP was introduced. The dryer was inserted into a dry box where an inert nitrogen atmosphere was maintained. Upon completion of drying and the removal of the dry WP granules from the dry box, ignition was instantaneous. All indications are that it is possible to mechanically dry WP granules; however, due to the inert atmosphere conditions, it does not seem feasible for production.

The use of chemical separation and drying of WP was attempted first with trichloroethylene. Phosgene gas was given off. The trichloroethylene reacted in some manner with the WP and the WP would not ignite. Freon 11 (trichloromonofluoromethane) was used with the water-WP system. Freon 11 and water are immiscible. Also, Freon 11 acts as a cooling agent, thus suppressing ignition of the WP. Freon 11 is denser than water and therefore the WP is trapped in the Freon layer and the water can be decanted off. The Freon-WP was mixed with the C-1 resin and WE resin. Freon was removed by evaporation. Nineteen rounds were filled with C-1 epoxy and 19 rounds with WE, H-25 epoxy. With the use of X-ray analysis of the munitions, it was hoped that a prediction of firing results would be possible.

With the information that the WP system could be water-free, it was suggested that polyurethane-WP system be tested. Using the same mixing procedure as used with the WP-epoxy system, PEG 400 and TDI were added to the Freon-WP system. The Freon 11 was evaporated at room temperature and the mix was extruded into six shells.

The results of the test firing with the previously mentioned shells were as follows:

Formulation	Firing temperature	Number of rounds on target/fired
C-1, temperature cycled and vibrated	130°F	18/23
C-1, immediate closure	130°F	7/23
WE, H-25, temperature cycled and vibrated	130°F	0/24
WE, H-25, immediate closure	130°F	0/10
C-1, vibrated only (within 3 days of fill)	130°F	3/8
C-1, dry WP with Freon 11	130°F	0/10
C-1, dry WP with Freon 11	55°F	5/5
WE, H-25, dry WP with Freon 11	130°F	0/9
WE, H-25, dry WP with Freon 11	55°F	2/5
Polyurethane, dry WP with Freon 11	130°F	0/6

The X-ray analysis of the closed munitions was not conclusive enough to make a successful prediction on the flight stability of the munitions. However, the rounds which had nonuniform voids and were predicted not to fly, did not fly.

The C-1, WP system's immediate closure rounds showed some interesting trends. Of the first eight rounds fired, seven functioned on target. The only difference between these rounds and the remaining 15 rounds (of which none functioned on target) was the time interval between loading and welding closed. The time difference between the loading and welding from the first to last rounds was 90 minutes to 30 minutes. This suggests that the welding has a negative effect on the WP-epoxy system when the epoxy has not had sufficient cure time.

With the results of the above test firing, it was decided to investigate the parameters of cure time before closing and cure time before vibration. The shells filled were as follows:

Formulation	Cure time before weld	Cure time before vibration	Number of rounds
C-1	12 hours	3 days	15
C-1	12 hours	Not vibrated	5
C-1	72 hours	6 days	15
C-1	72 hours	Not vibrated	5
C-1	72 hours	10 days	20
C-1	72 hours	12 days	18
Epon 828, V-40 cement filler	12 hours	3 days	10
C-1	72 hours	Not vibrated	2

Cure time before welding. Time between fill and welded closure.
Cure time before vibration. Time between fill and vibration tests.

All of the rounds were oven tested for 24 hours at 160°F for leakers after welded closure.

The results of the test firing for the rounds mentioned above were as follows:

Formulation	Cure time before weld	Cure time before vibration	Number of rounds on target/fired
C-1	12 hours	3 days	5/7
C-1	12 hours	Not vibrated	2/2
C-1	72 hours	6 days	8/14
C-1	72 hours	Not vibrated	3/7
C-1	72 hours	10 days	14/19
C-1	72 hours	12 days	8/10
Epon 828, V-40 cement filler			

All rounds fired in the above list had a reduced propellant charge in an attempt to duplicate the ballistic trajectory of the 40mm high explosive (HE) round.

Dr. E. O'Brien and Mr. D. M. Anderson, Picatinny Arsenal, suggested that a study of the exothermic reaction be made. The suggested study was carried out with various epoxy systems and various reaction conditions.

Test #1 was undertaken to determine if there is a measurable exotherm which can be evaluated with the use of thermocouples. This test was made using a constant water bath temperature of approximately 81°F. A thermocouple was placed in the bath so that the reaction conditions could be recorded at all times. The thermocouple results are plotted in Figure A-1 (appendix). A pure epoxy system was used for this test – Epon 828 and V-40 (versamide) in a ratio of three-to-one by weight. A 30-gram sample was placed in a 1.25-in diameter beaker with a thermocouple in the middle. The results of this test showed that there is a measurable exotherm in this reaction.

The same tests were repeated, using the variables previously mentioned. The plots of the thermocouple readings appear in Figures A-2 through A-5.

In Test #2, figure A-2 (line number 2) shows the thermocouple reading for a 50-gram sample of Epon 828 and V-40 in a 1.50-in diameter beaker. The results of this test indicated that as you increase the diameter of the test sample, the amount of heat from the exothermic reaction also increases. As part of Test #2 the exotherm from a 30-gram sample of C-1 resin and hardener was measured. The C-1 system, which contains approximately 68% by weight filler, demonstrated a higher exotherm than the Epon 828, V-40 system which had no filler. Therefore, it can be concluded that the C-1 system is not a versamide system as was originally believed.

In Test #3, figure A-3 (line number 2) shows the thermocouple reading for the Epon 828 and V-40 epoxy system with 68% by weight cement filler added to the V-40. This test was again run at an average cure temperature of 72°F; the exotherm was the same, if not larger, than that of the sample without filler under the same conditions. Also, as part of Test #3, figure A-3 (line number 3), a sample weighing 45 grams, containing 70% WP and 30% epoxy (Epon 828 and V-40 with 68% cement filler), demonstrated a slightly lower exotherm than the system without WP.

In Test #4, figure A-4, three tests were run simultaneously with a constant water bath temperature of 130°F. The first test (line number 2) was a 30-gram sample of Epon 828 and V-40. The maximum ΔT (difference between sample temperature and bath temperature) was 8°F. This indicates that as cure temperature is increased in a pure epoxy system, the epoxy cures at a faster rate and has a higher exotherm. The second test (line number 4) was 30-gram sample of the C-1 epoxy system in a 1.25-in diameter beaker. The maximum ΔT was 105°F. In relation to the previous tests with the Epon 828 and V-40 epoxy system, the results show that the exotherm is much greater when the cure temperature is increased. The third test (line number 3) was the same as the second, except that 1 ml of water was added. The maximum ΔT obtained was 5°F. Also, the cure time doubled for this sample. Therefore, it is concluded that water in an epoxy system retards cure, giving the epoxy a longer pot life, and that water decreases the exotherm to a point where, in a WP system, the WP would not melt. An additional test was made at this temperature with the C-1 epoxy (30%) and WP (70%) at a cure temperature of 130°F. However, no conclusive results could be made due to the fact that the WP settled out of the epoxy. This did indicate that some minimum cure time at ambient temperature is required.

In Test #5 (figure A-5), two runs were made with a cure temperature of 70°F. The first test (line number 2) was the C-1 epoxy system in a 1.50-in diameter beaker. The maximum ΔT obtained was 12°F. The same test was made using a 1.25-in diameter beaker (line number 3). The largest ΔT obtained was 10°F. It is therefore concluded that as the diameter of the vehicle is increased, the exotherm increases.

An extended test run was made on the C-1 epoxy system in a 1.25-in diameter beaker with a cure temperature of 70°F. The results were the same as in Test #5 and there was no appreciable exotherm after the initial one which has been demonstrated.

Another test firing was completed in January 1967 with both the standard HE propellant charge and a reduced propellant charge. The quantity of WP was also varied in these rounds and the results are as follows:

Formulation	Propellant	Number of rounds on target/fired
C-1, 70% WP, 30% C-1	Standard HE propellant charge	8/10
C-1, 50% WP, 50% C-1	Standard HE propellant charge	7/10
C-1, 50% WP, 50% C-1	Reduced propellant charge	10/10

Also included in this firing were 19 rounds of C-1 composition with 70% WP. Fourteen of these rounds were recovered, X-rayed, and sectioned. The X-rays showed that in a cylindrical area running through the center of the shell, there was a variation in density. However, when the rounds were sectioned, there was no visible variation between the 11 rounds which flew full range and those that fell short (less than 1500 yards).

The welded closure was not accomplished until after a 12-hour cure had been effected.

A series of tests was run to determine the curing time necessary to assure complete adhesion to the munition wall after the round had been filled. These tests were run on both the C-1 epoxy system and the R603 resin, H-36 hardener system. Results of these tests proved that 12 hours was necessary to completely cure the WP/epoxy mixture to prevent migration of the WP granules and to securely bond the material to the munition wall.

The erratic results of the firing tests led to speculation that at elevated temperatures (115°F to 130°F) the small pockets of WP in the matrix of epoxy resin were in a liquid state, and though they did not change position, vibrations within the small pools of WP caused the round to be ballistically unstable.

In order to prove or disprove this theory, it was decided to replace the WP in the epoxy with an inert, solid granule of the same mesh size and specific gravity as the WP. Anthracite coal was the material selected to simulate the WP granules. Twenty 40mm XM655 rounds were filled with coal/C-1 epoxy system, and twenty with coal/R603-H-36 epoxy system. The rounds were X-rayed and assembled with inert fuzes and burster pellets, temperature-conditioned to 130°F, and dynamically fired for range, accuracy, and recovery.

Ten of the forty rounds fired tumbled in flight and traveled less than 1100 meters. The rounds were recovered and examined. In all cases the fuze had either unscrewed several threads or fallen completely off. Since the fuze had never been cemented on in any of the previous tests, it was felt that this was the major factor causing ballistic instability and not vibration of molten WP, since these rounds contained no WP.

An additional test series was then planned to evaluate both the fuze theory and the C-1 epoxy system versus the R-603, H-36 system. Twenty-five rounds were filled with a WP/epoxy mixture of each of the above systems. The rounds were then X-rayed to detect any voids, oven-tested at 160°F and then assembled with live fuzes and burster pellets. However, this time the fuze was cemented to the round and allowed to cure for 24 hours. The rounds were then temperature-conditioned at 130° for 24 hours and were test fired. All of the 50 rounds traveled 1450 meters or more with less than two feet deviation.

Since the R-603, H-36 epoxy system was much easier to process than the C-1 system because of its lower viscosity, another test firing was planned. This test series was made as an additional check on both the

R-603, H-36 epoxy system and the fuze theory. Fifty empty 40mm XM655 bodies were subjected to moments of inertia and centrifugal balance tests; the results of these tests proved that the empty rounds were not uniform and none were in complete balance. A seven-gram lead weight was fused onto the inside wall of each of 20 rounds at the point where the base meets the side wall. This was done to drastically unbalance the rounds as a comparison with the normal round. The bodies were then filled with 70%/30% WP/epoxy mixture using the R-603, H-36 systems. They were then subjected to an oven leak test for 24 hours at 160°F. The fifty rounds were X-rayed and again subjected to moments of inertia and centrifugal balance tests. Other than the 20 rounds with the off-center weights, no significant difference was found in the rounds before and after filling. The test rounds were then assembled with live fuzes and burster pellets with the fuzes cemented in place.

The assembled rounds were temperature-conditioned for 24 hours at 130°F and were fired for range and accuracy. All of the rounds, including the 20 unbalanced with the lead weights, were ballistically stable and traveled 1450 meters or better with only minor deviation.

This test conclusively proved that the erratic rounds in the early tests were caused by the unsecured fuze assembly and not by the filling material. It also showed that the rounds filled with the R-603, H-36 epoxy system were equal to the C-1 epoxy system.

As a final check on the 40mm XM655 round filled with WP/epoxy mixture using the R-603, H-36 epoxy system, the following tests were conducted. Eighty rounds were filled with a mixture of 70% WP and 30% epoxy resin and were subjected to an oven leak test at 160°F for 24 hours. They were divided into four groups of 20 and submitted for the following rough handling tests:

Round number	Range, meters	Impact function	Remarks
		GROUP II	
V645	1525	OK	Good smoke display
V660	1610	OK	Good smoke display
V661	1650	Dud	Fuze failure
V662	1600	Dud	Fuze failure
V663	–	Air burst	Round burst at approximately 500-meter altitude
V664	1550	Dud	Fuze failure
V655	1575	OK	Good smoke display
V656	1600		
V657	1525		
V658	1550		
V659	1750		
V654	1550		
V653	1575		
V652	1650		
V651	1475		
V650	1550		
V649	1750	Dud	Fuze failure
V648	1550	OK	Good smoke display
V647	–	–	Round destroyed (bad fuze threads)
V646	1550	OK	Good smoke display

Range and accuracy reliability (19/20) of Group II = 95%

Round number	Range, meters	Impact function	Remarks

GROUP III

Round number	Range, meters	Impact function	Remarks
V563	1500	OK	Good smoke display
V564	1500		
V565	1575		
V566	1475		
V567	1425		
V570	1425		
V569	1575	Dud	Fuze failure
V568	1550	OK	Good smoke display
V571	1575	Dud	Fuze failure
V572	1575	OK	Good smoke display
V562	–	–	Round destroyed (bad fuze thread)
V561	1575	Dud	Fuze failure
V560	1550	OK	Good smoke display
V559	1525		
V558	1525		
V557	1525	Dud	Fuze failure
V556	1550	OK	Good smoke display
V555	1600	Dud	Fuze failure
V554	1450	OK	Good smoke display
V553	1600	Dud	Fuze failure

Range and accuracy reliability (19/19) of Group III = 100%

GROUP IV

Round number	Range, meters	Impact function	Remarks
V542	1200	Short round OK	Short round, good smoke display
V651	1350	OK	Short round, good smoke display
V639	1575		Good smoke display
V643	1575		
V644	1400		
V582	1525		
V581	1550	Dud	Fuze failure
V580	1500	OK	Good smoke display
V579	1550		
V578	1525		
V576	1550		
V577	1600		
V574	1525		
V575	1475		
V573	1575		
V583	1450		
V584	1500		
V585	1500		
V586	1600		
V587	1550		

Range and accuracy reliability (18/20) of Group IV = 90%
Range and accuracy reliability of 78 rounds tested = 95%

During the firing tests the ambient temperature was 10°F with a strong cross-range wind (25 mph) gusting up to 35 mph. This strong cross-range wind could account for the three short rounds.

Group I	Vibrated at -65°F and +155°F
Group II	Vibrated at +155°F
Group III	Vibrated at -65°F

The above three groups were tested in accordance with TECP Directive 700-700, "Combined Vehicle and Aircraft Schedule for Ammunition."

Group IV	Subjected to the following temperature cycle shock tests (no vibration): three hours at -65°F, three hours at +155°F

At the completion of the rough handling tests, the rounds were shipped to the Frankford Arsenal experimental range at Fort Dix, NJ. The rounds were temperature-conditioned at 130°F for 24 hours and then fired for range, accuracy, and display. Listed below are the results of these tests.

Round number	Range, meters	Impact function	Remarks
		GROUP I	
V665	1725	Dud	Fuze failure
V666	1750	OK	Good smoke display
V667	1700	OK	Good smoke display
V668	1700	Dud	Fuze failure
V633	1800	OK	Good smoke display
V489	1550		
V630	1800		
V631	1600		
V594	1600		
V595	1550		
V596	1750		
V597	1000	Short round	Large globules of fuze cement on cartridge; round had tube forced into gun
		OK	
V599	1550	OK	
V552	1750		
V601	1750		
V602	1750	Dud	Fuze failure
V632	1725	Dud	Fuze failure
V629	1750	OK	Good smoke display
V598	1725		
V593	1700		

Range and accuracy reliability (19/20) of Group I = 95%

During the development of the WP/epoxy system, the only resins and hardeners tested had been obtained from the Specialty Plastics Co. of Baltimore, MD. To prevent being limited to one supplier, samples of the following epoxy resins and hardeners were submitted to the Sadtler Research Laboratories, Inc., Philadelphia, PA, for analysis and comparison with similar products from other suppliers:

Resins	Hardeners
C-1	C-1
R-601	H-35
R-603	H-36
	H-37
	H-38
	H-39
	H-40

The samples were examined as films from 2.5 to 15 microns, using a Perkin-Elmer Model 137 infrared spectrophotometer. Sample resins C-1 and R-601 and hardener C-1 were separated from the filler by dispersion in a suitable solvent and centrifugation. The solvent layer was drawn off, the solvent removed and the clarified product re-examined as above. The spectra were used for comparison with the Sadtler Standard Spectra of Pure and Commercial Materials.

Results of the laboratory study:

1. Resins C-1 and R-603.

These samples have infrared absorption patterns similar to the following products:

Product	Manufacturer
Epon resins in general	
Particularly Epon resins 828 and 1031	Shell Oil Co.
Genepoxy M180	General Mills
Epi-rez 510 B-6-6792	Jones-Dabney
Erl-3794	Bakelite
Scotchcast Resin #5 Part A	Minnesota Mining Co.
Resin X-2633.2	Dow Chemical
Epotuf 37-130	Reichhold Chemicals

2. Resin 603.

The strongest bands of the absorption spectrograms of R-603 match those of calcium carbonate. The rest of the profile fits the bands of resin 601.

3. Hardener C-1.

This spectrogram shows a pattern very similar to that of Epon Curing Agent C-111 manufactured by Shell Oil Co.

4. Hardener H-35.

This hardener shows an absorption pattern which closely matches that of Shell's Epon Curing Agent U.

5. Hardener H-37.

The closest matches found in the Standard Collections for this agent were as follows:

Epon Curing Agent V-1 Et-6	Shell Oil
Versimid 401	General Mills

In order to prepare specifications for epoxy resins and hardeners, it was necessary to obtain complete and detailed analyses. The following two epoxy systems were selected for study:

Resins	Hardeners
C-1	C-1
R-603	H-36

The C-1 system is the most viscous, while the R-603, H-36 system is the most fluid. Samples of the above systems were submitted to the Sadtler Laboratories to be analyzed for the following: viscosity, filler identification, percent filler in resin, particle size of filler, thixotropic agent, and identification of any other material.

Results of Analyses:

Viscosity

Sample	Viscosity
Resin R-603	10,200 centipoises
Resin C-1	500,000 centipoises
Hardener H-36	59,000 centipoises
Hardener C-1	454,000 centipoises

Particle Size

	Microns
C-1 Resin	3 to 60
C-1 Hardener	3 to 60
Resin R-603	4 to 7
Hardener H-36	4 to 7

The submitted samples were found to be physical mixtures of the following commercially-available components:

Resin R-603

Epoxy Resin	50%
Filler	50%

Hardener H-36

Epoxy-based aliphatic amine hardener	55%
Filler	45%

C-1 Resin

Epoxy Resin	45%
Filler	55%

C-1 Hardener

Epoxy-based aliphatic amine hardener	60%
Filler	40%

No other additions were found in any of the samples submitted.

Total Solids

	Total Solids @ 105°F
Resin R-603	94.3%
Hardener H-36	88.8%
C-1 Resin	99.6%
C-1 Hardener	90.9%

Total Ash

	Total Ash
Resin R-603	34.9%
Hardener H-36	27.7%
C-1 Resin	39.1%
C-1 Hardener	53.9%

The ashes of resin R-603 and hardener H-36 were tested and found to be calcium carbonate and calcium oxide, the oxide being formed by ignition of the carbonate. The percent ash, therefore, is lower than the actual amount of filler present.

The percent resin or hardener was determined by dispersion in a suitable solvent or solvent system and centrifugation to remove the filler. This process was repeated until the filler was free of organic matter. Results are as follows:

Sample	Percent Organic
Resin R-603	50.2
Hardener H-36	53.7
C-1 Resin	62.1
C-1 Hardener	42.0

Infrared spectrograms were prepared on the samples as-received. The spectra were compared to those of the Sadtler Standard Spectra of Pure and Commercial Materials. The fillers in epoxy resin R-603 and hardener H-36 were found to be calcium carbonate, such as Allied Whiting sold by Akron Chemical Co. and calcene from Pittsburgh Plate Glass. Other suppliers of calcium carbonate filler are: Diamond Alkali, United Mineral and Chemical, Georgia Marble, and US Gypsum.

The fillers in C-1 resin and C-1 hardener are mixed aluminum-calcium and aluminum-calcium-magnesium-silicates; not silicon dioxide (silica). Among the suppliers are: Georgia Kaolin, Phillips Minerals and Chemicals, L.A. Solomon and Brothers, and American Colloid.

Comparison of spectrograms of epoxy resin R-603 and C-1 with those of the Sadtler Collection yielded the following compounds with closely matching spectra:

Epoxy Resin C-1

Compounds	Supplier
Genepoxy M180, 175, 177, 190	General Mills
Epon 830 and 826	Shell Chemical Co.
Epi-Rez 510 B-6-6792	Jones-Dabney

Scotchcast Resin No. 2	Minnesota Mining and Mfg.
Rp-1130A	Ren Plastics
Resin X-2633.1 and X-2633.2	Dow Chemical Co.
Epocast 2D	Furane Plastics
Epotuf 37-128 and 37-140	Reichhold Chemicals

Epoxy Resin R-603

Epon 1310 and 1031	Shell Chemical Co.
Resin 2638.2	Dow Chemical Co.
Experimental Resin X-2638.3	Dow Chemical Co.
Avaldite 502	Ciba Corporation
Er 1-3794	Bakelite Corporation
Scotchcast Resin No. 5	Minnesota Mining & Mfg.

Spectrograms of H-36 and C-1 hardeners were virtually identical to the following products:

Epon Curing Agent U, C-11, and T 1	Shell Chemical Co.
Hardener 180	Applied Plastic Co.
Er 1-2807	Bakelite Co.
22LD-D-0814	Bakelite Co.
Hardener 3260	Ren Plastics

Results of the Sadtler Laboratories analysis showed that the R-603, H-36 epoxy system was far less viscous than the C-1 system. Past tests conducted to determine time required to obtain a homogeneous mixture of WP/epoxy showed that fluidity was of the epoxy system and was the determining factor. Therefore, the R-603, H-36 epoxy was selected in the system around which a continuous-processing plant would be designed.

As a final check on the ballistic stability and display of WP/epoxy when compared to straight WP, the following series of tests was conducted.

40mm XM655 Dynamic Firing Tests

Eighty rounds were filled with 70%/30% mixture of WP/epoxy, using the R-603, H-36 system. The 30% epoxy consisted of 22% resin and 8% hardener. The rounds were allowed to cure 12 hours after which they were welded closed and leak-tested at 160°F for 24 hours.

An additional eighty rounds were filled with straight WP and were handled in the same manner as described above. After the rounds had been leak-tested, they were temperature-conditioned for 24 hours at 130°F and were fired at this temperature for distance and accuracy on a 1500-meter range.

All of the WP/epoxy rounds traveled 1450 meters or better with a maximum of one foot deviation. However, the standard WP rounds either tumbled and fell short or curved 50 to 100 meters off course.

105mm M416 Static Firing Test

The following static firing test was made to evaluate the smoke display of the 105mm M416 round filled with a 70%/30% WP/epoxy mixture with that of a standard WP-filled round. A series of nine static firing tests was made. Each test consisted of simultaneously firing two WP/epoxy rounds and one standard WP round. The rounds were placed in a straight line, 100 feet apart, with a standard WP round in the center. The 27 rounds used in this series were divided into three groups with nine rounds in each group. Each group was fired under different temperature conditions according to the following schedule:

Test number	Round Arrangement
1, 2, 3	LP - LS - LP
	HP - HS - HP
	AP - AS - AP

LP	Low temperature polymer @ -65°F
HP	High temperature polymer @ +145°F
AP	Ambient temperature polymer @ +40°F
LS	Low temperature standard WP @ -65°F
HS	High temperature standard @ +145°F
AS	Ambient temperature standard @ +140°F

The above rounds were temperature-conditioned 24 hours prior to firing. All of the test items functioned with excellent smoke display and with very little difference between the standard WP and the WP/epoxy rounds. The tests were recorded on both high-speed and standard color motion picture film.

105mm M416 Dynamic Firing Test

This dynamic firing test program was conducted at the direction of Picatinny Arsenal in accordance with a test procedure prepared by the Artillery Ammunition Laboratories of Picatinny Arsenal. The following procedure was used for the tests, which were conducted at Yuma Proving Ground, AZ.

Test No.	Range (meters)	Temperature	Number of rounds
1	1,000 direct fire	-65°F	10 WP/epoxy
2	1,000 direct fire	+70°F	10 WP/epoxy
3	1,000 direct fire	+145°F	10 WP/epoxy
4	2,000 direct fire	+145°F	10 WP/epoxy
5	4,000 direct fire	+145°F	10 WP/epoxy
6	Maximum range 7,000 meters, maximum elevation indirect fire	+145°F	30 WP/epoxy

A standard WP and high explosive plastic (HEP) round was fired alternately with each of the above WP/epoxy rounds. A total of 240 rounds were fired during this test period with the following data recorded:

Muzzle velocity
Chamber pressure
Range
Deflection
Meterological conditions
Time of flight
Flight characteristics
Smoke cloud pattern and duration
Observers' comments

Results of these dynamic firing tests proved that the WP/epoxy rounds had the same ballistic stability and accuracy as the HE rounds at all ranges, and with a display comparable to the standard WP round. Details on individual rounds appear in a test report prepared by the Artillery Ammunition Laboratory, Picatinny Arsenal, NJ.

B. Development of a Prototype WP/Epoxy Continuous Processing Plant.

Continuous WP granulation equipment was designed, fabricated, tested, and found to continuously produce WP granules ranging in size from 14 to 30 mesh. A complete process study was conducted and the following recorded: flow rate and temperature of hot and cold water, lowest possible liquid WP temperature, pressure and flow rate of WP through nozzle, highest possible production rate, and material balance between WP charged to melting pot and percent recovery of WP granules.

In the investigation of WP/epoxy mixing and filling equipment, a series of tests was made to determine the metering qualities of the various resins and hardeners. Results of these tests proved that, with the exception of the C-1 system, all of the other resins and hardeners could be easily transferred and metered with excellent reproducible results.

A study was then made to determine which of the resin systems would produce the most homogeneous WP/epoxy mixture with the shortest mixing time. As a result of this study, the R-603 resin with the H-36 hardener was selected as the system around which the continuous mixing system would be designed.

Prior to fabricating a prototype pilot plant, a comparative study was made to evaluate a fully-continuous versus a semi-continuous system.

This study revealed that a semi-continuous system would be much easier to develop and operate if it were possible to make and store a large quantity of a mixture of resin and WP granules for a period of time without a change in viscosity or WP granule migration. This was determined by preparing one gallon of a mixture of R-603 resin and WP granules and allowing it to stand. After three months, no change occurred in the mixture so a semi-continuous mixing system was designed and fabricated.

In this process, dewatered copper-coated WP granules would be mixed with epoxy resin and stored. A duplicate storage tank would contain the hardener. These two tanks would feed two Moyno metering pumps regulated to deliver proportioned streams to a mixing and filling head. A flow diagram of the pilot system is shown in the figure.

The prototype pilot system has been completely fabricated and has been tested with simulants. However, no process studies with WP-epoxy have been conducted.

III. DISCUSSION.

The prime purpose of this program was to develop a WP/polymer system that would remain solid over the accepted military operating temperatures – thus avoiding the liquid phase at temperatures above 115°F which caused serious ballistic instability.

Final dynamic ballistic tests on rounds ranging from 40mm to 105mm proved that the WP/epoxy system developed under this program met all military temperature and ballistic requirements.

IV. CONCLUSIONS.

In order to meet the military requirements of an encapsulated white phosphorus, it was necessary to develop not only the WP/epoxy formulation but the equipment and procedures for processing the material and filling the various munitions. The program described in this report resulted in the following accomplishments:

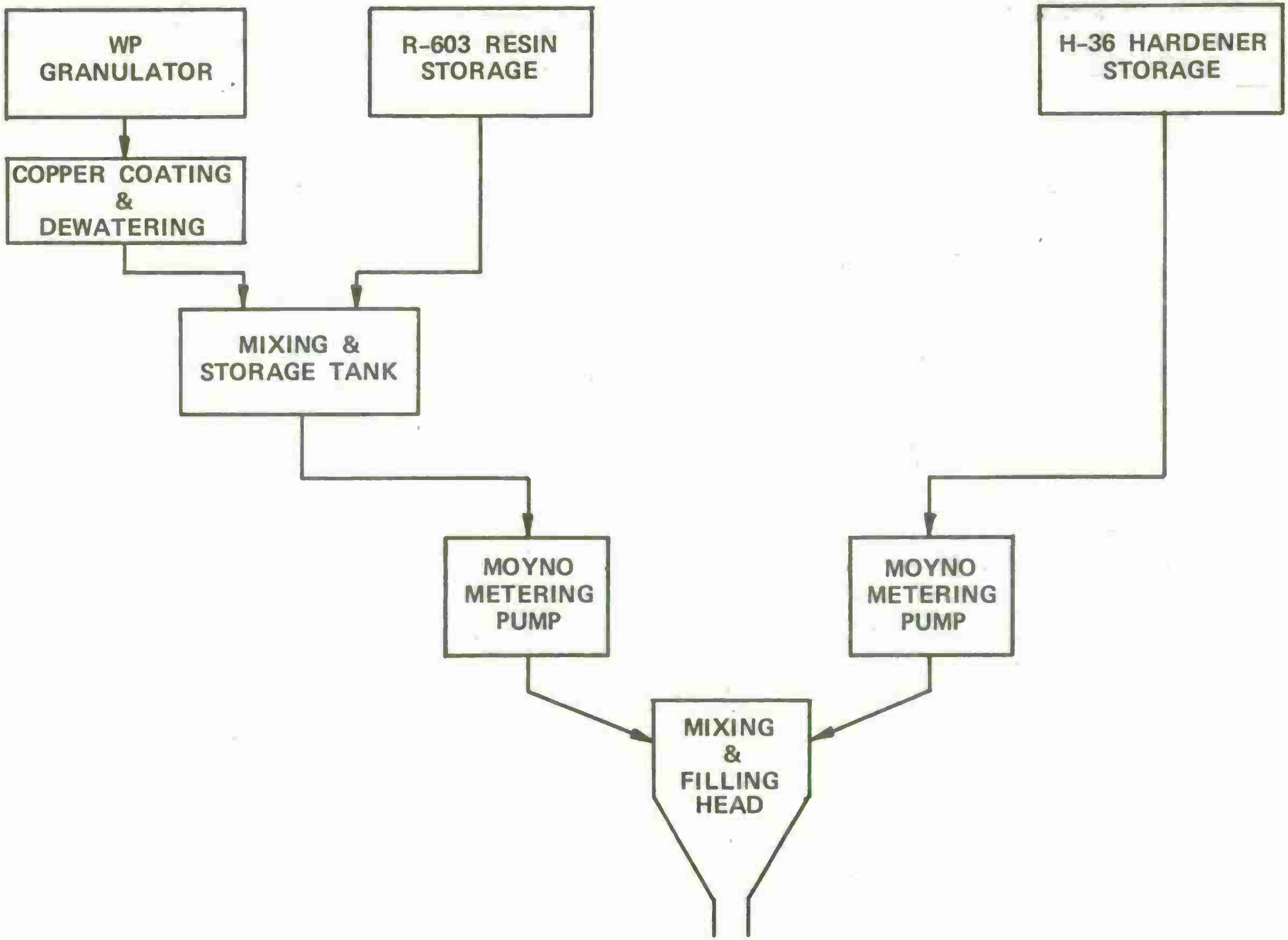

Figure. Flow Diagram of Pilot System for Semi-Continuous WP/Epoxy Mixing

1. An encapsulated white phosphorus formulation was developed and tested which met all military ballistic requirements over the complete operating temperature range.

2. Equipment was designed, fabricated, and tested which continuously granulated white phosphorus in particle sizes required by the formulation.

3. A variety of epoxy resins and hardener samples were obtained and tested. As a result, ten commercially-available products were found which met performance requirements.

4. A laboratory-size WP/epoxy pilot plant was designed and fabricated to continuously process material and fill munitions.

V. RECOMMENDATIONS.

A. Complete process studies should be conducted on the laboratory pilot plant to determine and correct processing problems.

B. Based on the results of the process studies, the design for a full-scale pilot plant should be finalized.

C. A full-scale pilot plant should be fabricated and tested.

EXOTHERM REACTION STUDIES

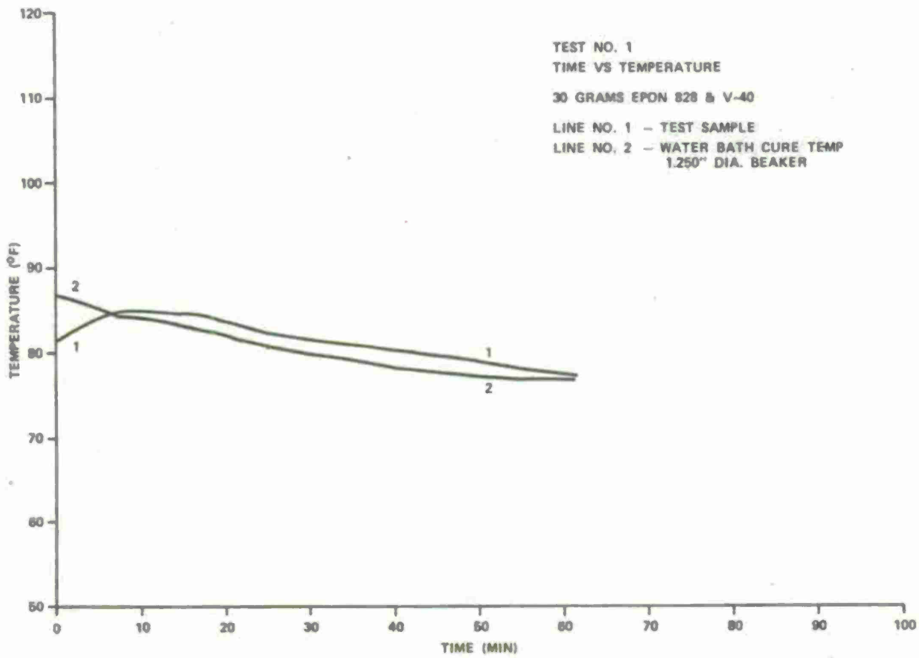

Figure A-1

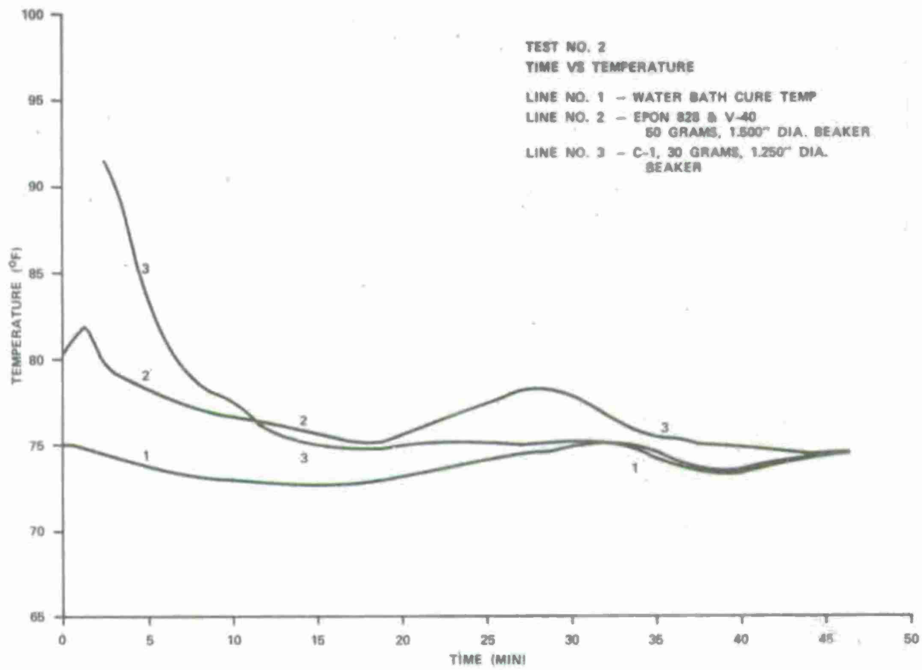

Figure A-2

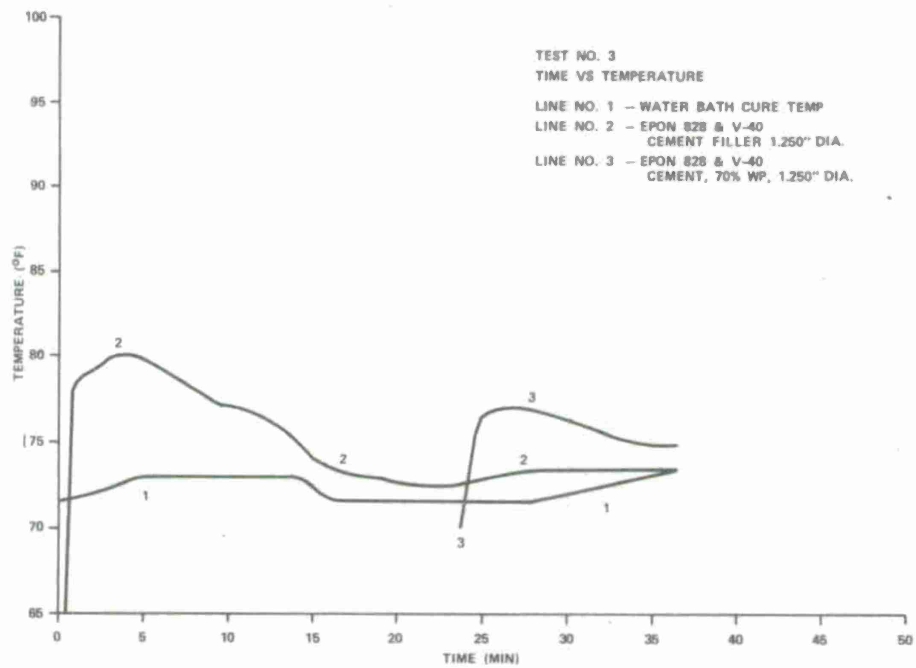

Figure A-3

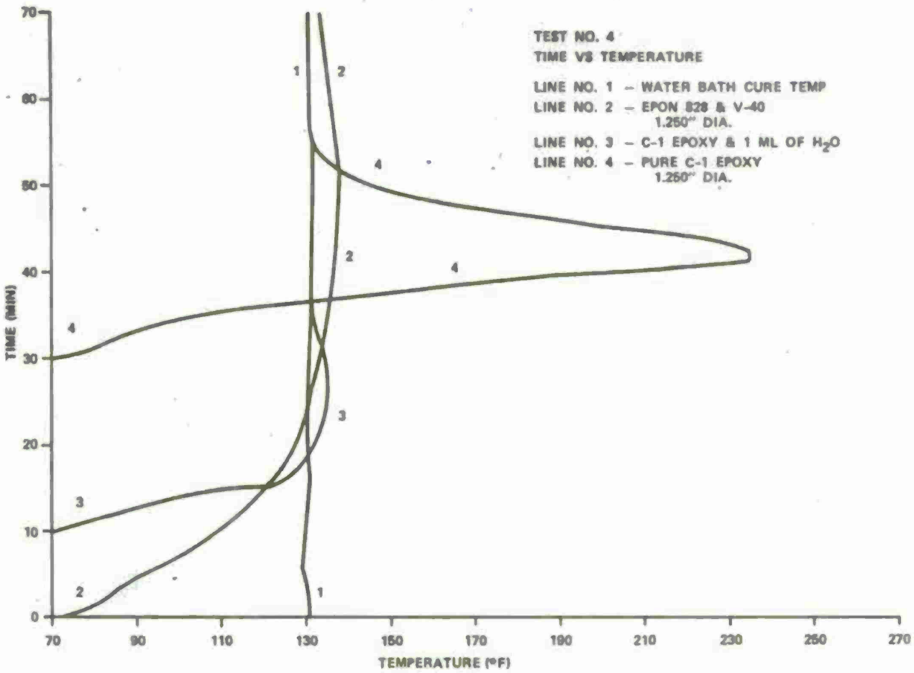

Figure A-4

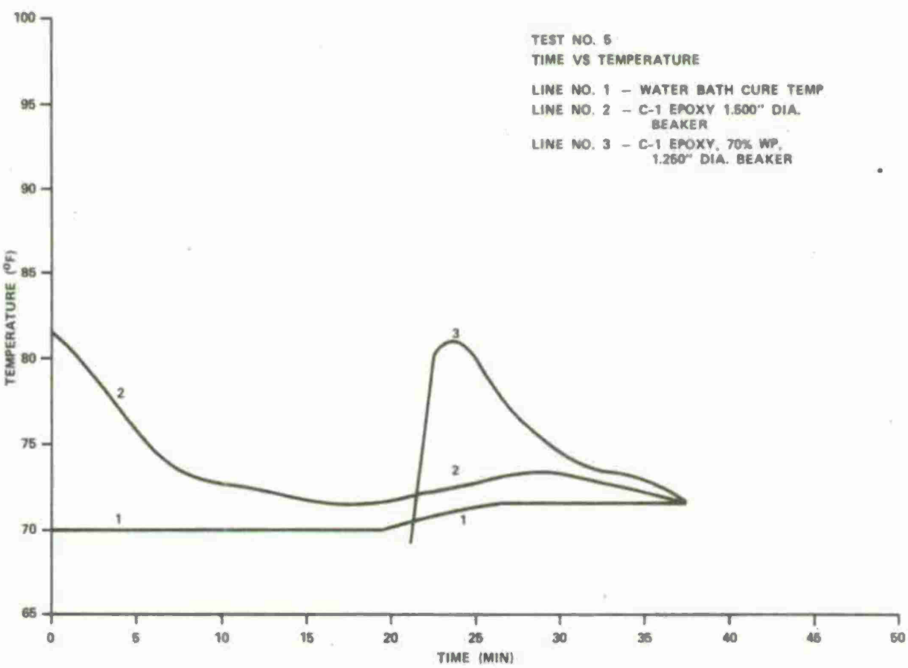

Figure A-5

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